



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of : Confirmation No.1988  
Masami KUJIRAI : Attorney Docket No. 2004-1547A  
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WATER-BASED HEAT-RADIATION-PREVENTIVE COATING MATERIAL  
FOR GLASS, HEAT-RADIATION-PREVENTIVE GLASS, AND  
METHOD OF PREVENTING HEAT RADIATION

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DECLARATION UNDER 37 CFR 1.132

Sir:

I, Yoshihito SAKURAI, declare as follows:

I. IDENTIFICATION OF DECLARANT

I am employed by Suikoh Top Line co., Ltd. and hold the position of associate manager of Department of Technical Development.

My educational background is as follows:

Graduated from Meisei University  
Doctor's Degree in Chemistry

II. DETAILS OF EXPERIMENTS

I have conducted personally or under my direction and control the following experiments [1] and [2]:

**Experiment [1]:**

1. Purpose of Experiment

The purpose of the following experiment [1] is to show that, if the coating material contained porous inorganic metal

oxide such as porous silica disclosed in Scholz et al. (US 5,997,621) is used, heat-radiation-preventive effect of the present invention cannot be obtained.

## 2. Preparation of Sample(1) and Sample(2):

### <Sample(1)>

0.2% by weight of N-( $\beta$ -aminoethyl)  $\gamma$ -aminopropyl triethoxy silane, trade name;"KBM603", manufactured by Shin-Etsu Chemical Co., Ltd., was dissolved in deionized water having the total anion content of 65.7 mgCaCO<sub>3</sub>/L to prepare a coating solution.

The coating solution thus prepared was coated onto one side of a glass plate of 50cm  $\times$  50cm  $\times$  3 mm-thickness by means of using Meyer bar No. 6 and then the glass plate was dried enough at 100°C in a fixed-temperature dryer bath to obtain Sample(1).

### <Sample(2)>

Firstly, an aqueous solution of colloidal silica stabilized by Na (trade name; "Snowtex S", manufactured by Nissan Chemical Industries, Ltd., silica content; 30% by weight, particle diameter; 8-11 nm, pH9.5-10.5) was diluted by deionized water having the total anion content of 65.7 mgCaCO<sub>3</sub>/L to obtain a diluted aqueous solution having 5% by weight of silica concentration.

Then, 0.2% by weight of N-( $\beta$ -aminoethyl)  $\gamma$ -aminopropyl triethoxy silane, as a silane coupling agent, was added and dissolved into the above-obtained diluted aqueous solution to prepare a coating solution.

The coating solution thus prepared was coated onto one side of a glass plate of 50cm  $\times$  50cm  $\times$  3 mm-thickness in the same manner as in the case of above-mentioned Sample(1) to obtain Sample(2).

## 3. Evaluation

Three same boxes with the outer size of 50cm by 50cm with a height of 50cm made from styrene foam wherein one side was open were prepared.

Above-mentioned Sample(1), Sample(2) and a glass plate without coating were adhered by silicone sealant to the open side of the boxes made from styrene foam so that, in the cases of Sample(1) and Sample(2), the side of the glass plate wherein the coating solution was applied faces to the inside of the boxes, and thus test bodies were prepared.

The test bodies were installed in a wide place where the test bodies can be irradiated by sunlight well. The opening side of the test body wherein the glass plate was attached was directed upwards.

The inside temperature of the test bodies was determined when said temperature comes to equilibrium after exposed to sunlight. The outside air temperature at this time was 31.9°C. The results were shown in the following Table A.

Table A

Test Samples	Equilibrium Temperature of the Box inside (°C)	Outside Air Temperature (°C)
Glass plate without coating	52.3	31.9
Sample(1)	50.1	31.9
Sample(2)	53.2	31.9

#### 4. Result

The results of the experiments shown in the above Table A indicate that the internal temperature of the box using Sample(1) comprising only a silane coupling agent and deionized water became lower by 2.2 °C than that using a glass plate without coating, whereas the internal temperature of the box using Sample(2) comprising porous silica in addition to a silane coupling agent and deionized water became higher by 0.9°C than that using a glass plate without coating.

The foregoing experiments demonstrate that heat-radiation-preventive effect does not exhibit in the case of using a coating film comprising porous silica. As a matter of fact, the glass plate having a coating film comprising porous silica exhibited heat-retaining effect, and therefore the internal temperature of the box became higher. It is probably because that heat conductivity of the glass plate became lower by coating a solution comprising an inorganic metal oxide sol capable of forming a porous inorganic metal oxide network. The porous inorganic metal oxide network has low heat conductivity because of the presence of air in the pores. The glass plate having low heat conductivity exhibits high heat insulation. The internal temperature of the box having a glass plate of high insulation would increase because of its heat-retaining property. This is the reason why the internal temperature of the box having the glass plate coated with a solution comprising an inorganic metal oxide became higher.

Thus, it is clear that a coating material contained inorganic metal oxide would materially affect the basic nature of the coating material of the present invention.

### **Experiment [2]:**

#### **1. Purpose of Experiment**

The purpose of the following experiment [2] is to show that, if the coating material contained a tetrafunctional silane compound disclosed in Havey et al. (US 6,001,163) is used, heat-radiation-preventive effect of the present invention cannot be obtained.

#### **2. Experiment [2]-1**

5% by weight of tetraethoxysilane otherwise known as tetraethyl orthosilicate,  $\text{Si}(\text{OC}_2\text{H}_5)_4$ , trade name, "KBE-04", manufactured by Shin-Etsu Chemical Co., Ltd., was mixed with 95% by weight of deionized water having the total anion content

of 65.7 mgCaCO<sub>3</sub>/L with stirring.

However, the liquid phase of the mixture thus obtained was separated. That is, the mixture could not form a uniform solution which means that the mixture can not form a coating film.

### 3. Experiment [2]-2

1% by weight of N-(β-aminoethyl) γ-aminopropyl triethoxy silane, trade name;"KBM603", manufactured by Shin-Etsu Chemical Co., Ltd., was dissolved in deionized water having the total anion content of 65.7 mgCaCO<sub>3</sub>/L to prepare a coating solution.

Subsequently, 5% by weight of tetraethoxysilane, trade name;"KBE-04", manufactured by Shin-Etsu Chemical Co., Ltd., was mixed with said coating solution.

However, the liquid phase of the mixture thus obtained was separated and it was not possible to form a uniform solution and a coating film thereof.

### 4. Experiment [2]-3

1% by weight of 3-glycidoxypropyl-trimethoxysilane, trade name;"KBM-403", manufactured by Shin-Etsu Chemical Co., LTD., was dissolved in deionized water having the total anion content of 65.7 mgCaCO<sub>3</sub>/L to prepare a coating solution.

Subsequently, 5% by weight of tetraethoxysilane, trade name;"KBE-04", manufactured by Shin-Etsu Chemical Co., Ltd., was mixed with said coating solution.

However, the liquid phase of the mixture thus obtained was separated and it was not possible to form a uniform solution and a coating film thereof.

### 5. Result

The results of the experiments shown in the above Experiments [2]-1 to [2]-3 indicate that when tetrafunctional silane such as tetraethoxysilane and deionized water can not form a uniform solution because of it is easily hydrolyzed.

In Havey et al., since tetrafunctional silane is an essential component, it is necessary to use an aqueous organic solvent such as low alcohol in order to control hydrolysis.

On the other hand, tetrafunctional silane can not be used in the present invention because, as shown in Experiments [2]-1 to [2]-3, a uniform solution can not be obtained and thus a coating film can not be formed.

Thus, it is clear that a coating material contained tetrafunctional silane would materially affect the basic nature of the coating material of the present invention.

### III. CONCLUSION

The foregoing experiments demonstrate that a coating material comprising porous silica according to Scholz et al. or a coating material comprising tetrafunctional silane according to Havey et al. can not achieve heat-radiation-preventive effect.

That is, the coating film for glasses of the present invention consisting essentially of a silane coupling agent represented by the general formula (I) and deionized water having a total anion content of 700 mgCaCO<sub>3</sub>/L or lower can achieve unexpectedly remarkable heat-radiation-preventive effect. Therefore, the present invention is not obvious over any of Scholz et al. and Havey et al.

### IV. VERIFICATION CLAUSE

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: Sept. 12, 2008 Signature: Yoshihito SAKURAI  
Yoshihito SAKURAI